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THE DEVELOPMENT OF A METHOD OF PRODUCING ETCH RESISTANT WAX PATTERNS ON SOLAR CELLS

FINAL TECHNICAL REPORT

MOTOROLA REPORT NO. 2365/4

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JPL CONTRACT NO. 955324

PREPARED BY

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THE JPL LOW-COST SOLAR ARRAY PROJECT IS SPONSORED BY THE U.S. DEPARTMENT OF ENERGY AND FORMS PART OF THE SOLAR PHOTOVOLTAIC CONVERSION PROGRAM TO INITIATE A MAJOR EFFORT TOWARD THE DEVELOPMENT OF LOW-COST SOLAR ARRAYS. THIS WORK WAS PERFORMED FOR THE JET PROPULSION LABORATORY, CALIFORNIA INSTITUTE OF TECHNOLOGY BY AGREEMENT BETWEEN NASA AND DOE.

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ABSTRACT

A potentially attractive technique for wax masking of solar cells prior to etching processes was studied. This technique made use of a reuseable wax composition which was applied to the solar cell in patterned form by means of a letterpress printing method. After standard wet etching was performed, wax removal by means of hot water was investigated.

Application of the letterpress wax printing process to silicon was met with a number of difficulties. The most serious shortcoming of the process was its inability to produce consistantly well-defined printed patterns on the hard silicon cell surface.

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1.0 SUMMARY

This project studied the use of printed wax to serve as etch resistant patterns in the low cost manufacture of silicon solar cells. The use of printed wax suggested several advantages over the presently used techniques in terms of economy and reduced processing time. In addition, removal of wax from processed cells and its subsequent reuse afforded the possibility of further economy along with the reduction of waste material output.

A study of four techniques (transfer, lithography, intaglio and letterpress) identified letterpress printing as having the fewest problems where applied to solar cell processing. The most overall suitable masking wax identified was an Apiezon W solution. The suitability of Apiezon W as a masking agent was demonstrated on bare silicon, and on silicon coated with silicon dioxide and with silicon nitride, both textured and planar. Various methods of wax removal were researched, with emphasis placed on a system that avoided the use of organic solvents. Although removal by boiling water or steam appeared promising, complete removal was not possible by either of these methods.

The net result of the project revealed that letterpress printing produced less than satisfactory image quality for use in solar cell processing, and that complete removal of wax from processed wafers by means of water or steam is not practical.

2.0 INTRODUCTION

The patterned conductors on silicon solar cells may be incorporated into the process sequence in one of three ways:

1. Metal deposition on a bare silicon surface
   (a) Evaporate metal pattern through a mask
   (b) Silk screen metal pattern
   (c) Evaporate (or sputter) metal over the entire surface, and incorporate a separate patterning step.
2. Metal deposition in a pattern on the dielectric (AR) layer, and reaction through the AR layer to contact the silicon.
3. Metal deposition in patterns opened in a dielectric (AR) layer.

Considering the long range DOE price projections, methods 1 and 2 are considered to be too expensive in terms of basic materials cost and/or processing cost. Method 3 can incorporate a simple and economical base metal plating technology; it is necessary, however, that technology exist for providing the openings in the pre-applied dielectric (AR) coating in an economical manner.

One dielectric patterning technique, commonly used in the semiconductor industry, involves photoresist. A photosensitive liquid, known as photoresist (PR) , is applied to the cell surface by spraying or rapid spinning as a thin layer of controlled thickness. As the freshly applied PR layer is viscous and sticky at this point, a baking step is performed to drive off residual solvent and produce a surface that permits further processing. After baking, the PR layer is exposed to UV light through a shadow mask, irradiating the layer in the intended pattern. The irradiation promotes chemical reactions in the PR layer, causing the illuminated areas to be either more or less soluble than the unilluminated portions, depending on the type of PR used (positive or negative). The cells, after an additional bake, are exposed to a solvent which selectively removes photoresist, leaving behind a PR pattern resembling that on the shadow mask. The remaining pattern is used to protect parts of the device from the etchant agents that are employed to dissolve away the unmasked dielectric (e.g., silicon dioxide or nitride) layers. After etching, the PR must itself be removed by a special stripping solution.

The use of photoresist presents a number of problems. The material itself is expensive. Being light sensitive, PR processing areas must be illuminated by yellow safety light and must also be carefully temperature- and humidity-controlled. The photographic process by which the PR is patterned is time consuming, and calls for expensive exposure equipment and carefully prepared photographic glass plates bearing the intended pattern image (or its photographic negative). Moreover, the PR mixtures available are non-reuseable. Once applied and processed, the PR undergoes irreversible chemical alteration and must be chemically destroyed in order to effect removal from the substrate surface. Other disadvantages are also encountered. PR contains organic solvents, such as xylene, which are released to the atmosphere during use; some PR mixtures generate gas during storage, causing potentially dangerous pressure buildup; and virtually all have limited shelf life.

The use of wax as a substitute for photoresist results in substantial benefits. Wax is generally less costly than carefully formulated PR preparations. Unlimited shelf life is possible, since wax, either in pure solid form or as a mixture with an organic solvent, is relatively insensitive to temperature and does not depend for its effectiveness on photochemical reactions. Even if solvent-wax mixtures are used instead of pure wax, the use of organic solvents to develop the image is avoided, thereby lessening the amount of solvent released to the atmosphere.

Processing time is significantly reduced by avoiding the lengthy multiple baking cycles and exposure steps necessary with photoresist. To the above must be added the lack of a need for a physically separate photoresist facility requiring special illumination, humidification, and temperature control.

Since wax is not light-sensitive, its use as a patterned masking agent requires a method of producing a pattern without the exposure or developing processes involved with the use of photoresist. Any of various printing methods suggest themselves as suitable for forming the required pattern. Silk-screen printing has been successfully employed for this purpose. Silk screening requires the use of properly prepared formulations which have appropriate flow properties while maintaining useful etch resistance consistent with ease of removal. The purpose of this program was to investigate some form of impact printing, using a printing device similar to an ordinary office stamp, which would provide benefits of simplicity, coating durability, and speed over current silk-screen methods.

The project entailed the performance of several tasks:

1) Determine the printing method
2) Determine a suitable wax resist
3) Text printing results and modify process if necessary
4) Evaluate methods of wax removal.

3.0 TECHNICAL DISCUSSION

3.1 TYPES OF PRINTING

Four printing variations were studied: transfer printing, lithography, intaglio, and letterpress.

3.1.1 TRANSFER PRINTING

Transfer printing involves a process analogous to the use of carbon paper or a film typewriter ribbon. A thin, durable film carries upon it a thin l yer of wax which is transferred to the solar cell substrate surface by pressure applied to the back of the film. If pressure is applied in a pattern, a patterned layer of wax is transferred.

It was suspected that this method of printing would be the least desirable; experimental results bore this out. Too thick a wax coating on the transfer film caused either failure to transfer or ragged line edges where the wax had torn away from the supporting film. Thinner wax coatings improved edge definition but resulted in spotty wax coatings with pinholes and relatively large areas of unmasked surface. In addition, adhesion was found to be poor, with the thin wax coating lifting off the substrate during etching. This method was rejected.

3.1.2 LITHOGRAHY

In lithography, a flat surface is treated so that parts of it attract and hold a substance such as ink or wax, while other parts are repellant. The definition of attractive and repellant areas is achieved by a number of methods, such as application to an area of the plate of a special grease, or plating certain areas with a metal to which wax adheres, while the plate is composed of a repellant material.

The preparation of lithographic plates proved to be both complicated and expensive. The use of a grease pattern to define the wax-bearing areas of the plate was not practical since the grease image would wear off very quickly in use or suffer rapid image degradation due to the grease solvent properties of the masking wax.

The use of wax-repellant and wax-attractive regions on metal printing plates appeared practical at first. In practice, the original pattern was transferred from an existing photomask to plastic film, which is required in order to expose a photoresist coated metal plate, and the developed plate was then plated with a wax-attractive coating of copper. After plating, the photoresist was stripped and the plate was ready for use.

The expense of these items was substantial. Production of a film image of the pattern cost \$6, stainless steel cost \$2 and photoresist treatment of stainless steel plates cost an additional \$8. Expenses of approximately \$10 were required to copper electroplate the steel plate and strip photoresist. The total cost per plate was thus \$26.

This cost however, was not a one-time expense. Deterioration of the printing plate, as a result of scratching and/or wearing of the soft copper plated region required its periodic replating after 100 to 200 cycles. Even though the stainless steel plate itself could be reused, the plating process required reapplication of photoresist and patterning, costing \$18 each time, or 9 - 18¢ per substrate. In view of the cost the lithographic method was dismissed as being impractical.

3.1.3 INTAGLIO

Also originally planned for study was intaglio printing, in which wax is borne by depressions in a plate, the raised plate areas being resistant to wax wetting. Surface tension of the wax causes "beading" of the wax in the depressions, resulting in wax protruding above the raised plate areas (Figure 1). Trials of this technique showed that the masking wax had insufficient surface tension to cause the required beading. In addition, a durable wax antiwetting agent for the raised plate portions was not found. Stray droplets of wax on raised plate portions also contributed to the problems associated with this method. In view of the impracticalities involved, the intaglio technique was dropped from further consideration.

3.1.4 LETTERPRESS

Letterpress printing makes use of a plate that has protrusions in those areas that are to receive wax. Wax is applied, usually by roller, and is deposited mainly on the protrusions. The plate is then pressed against the substrate, where the wax borne by the protrusions transfers to the substrate (Figure 2).

Preliminary trials of letterpress wax printing confirmed its superiority to the other types of image production techniques and it was chosen for use in the remainder of the project.

3.2 PRINTING METHOD

The low-cost production of printing plates was essential to the success of the project, and methods were developed to produce large quantities of plates inexpensively.

A master mold was prepared, by photoresist and wet acid etching processes, of the intended image. The mold took the form of a negative image of the printing plate, with depressions in the areas where protrusions were desired on the plate. In this way printing plates could be produced of any curable liquid material by casting the liquid in the master mold and removing the material in the form of a finished plate after hardening (Figure 3).

FIGURE 1

INTAGLIO PRINTING TECHNIQUE

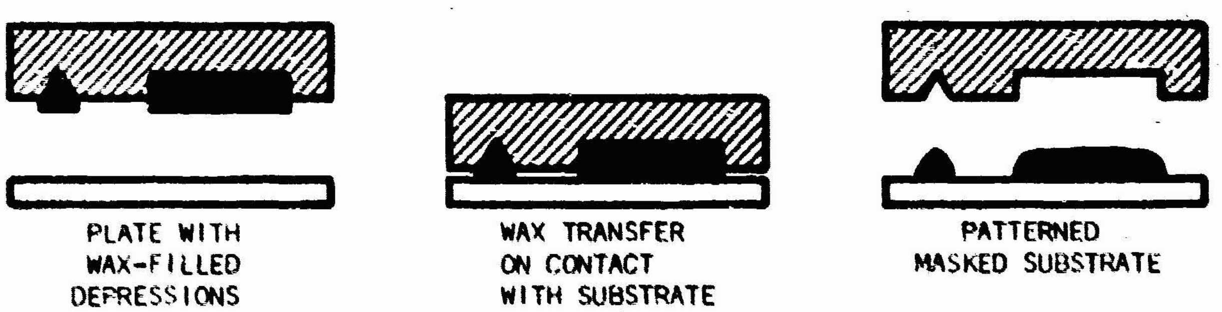


FIGURE 2

LETTERPRESS PRINTING TECHNIQUE

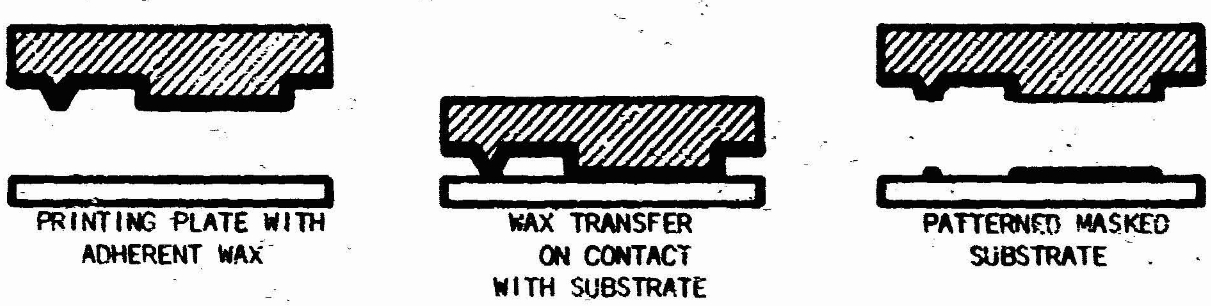
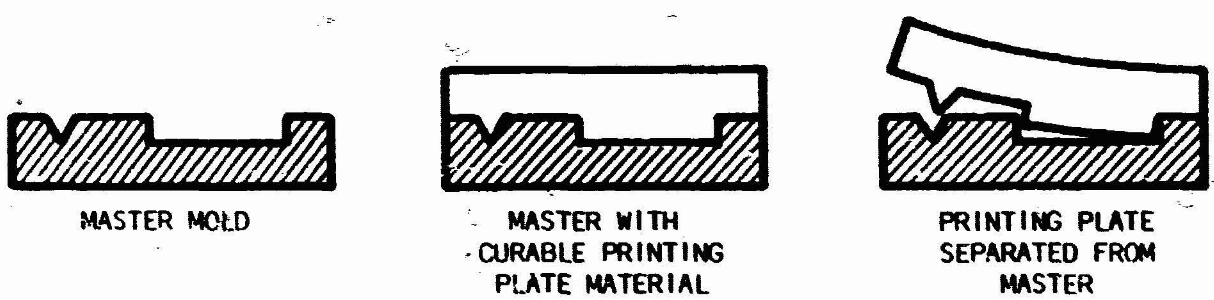


FIGURE 3

PRINTING PLATE PRODUCTION



In order to find the master mold material capable of combining the desirable qualities of physical durability and the capability of accepting detailed etching, molds were produced of several materials: aluminum, copper, glass, Kovar, and stainless steel. Experimentation showed that glass was unacceptable as a mold material - aside from its fragility, it failed to etch deeply and displayed an uneven etch rate which made the attainment of fine-line image features impossible. Aluminum also proved less than ideal because of its softness. Aluminum also shared a common defect with copper - both metals were attacked by acids present in some of the materials from which plates were molded, particularly the RTV silicone rubbers. Stainless steel and Kovar molds proved the most detailed and durable.

Materials used for printing plate production were varied. The project originally sought to employ molten wax as a masking agent, requiring the use of plate materials which were capable of withstanding sustained elevated temperatures of approximately 150°C. It was later determined, for reasons discussed in Section 3.4, that room temperature printing with solvent-wax solutions would be attempted in place of the hot wax printing. This widened the choice of printing plate materials. Those materials tested included: Sylgard 184 (a two part curable silicone), castable acrylic, plaster, portland cement, epoxy, vinyl, and polyethylene. Most of these materials failed for four basic reasons. 1) Brittleness caused crumbling of parts of the protrusions on the plaster and cement plates. 2) Excessive hardness prevented plates composed of acrylic and epoxy from contacting the wafer. 3) Absorption of solvent from solvent-wax solutions caused unacceptable swelling of acrylic, epoxy, and vinyl plates. 4) Excessive wear during use caused the rejection of polyethylene as a suitable material. This left Sylgard 184 as the preferred material. Sylgard is a two-part silicone rubber composition which is a pourable, self-levelling liquid when freshly prepared. The low viscosity of the liquid allows it to fill small features in the master mold and to relieve itself of any entrapped air bubbles. After standing for several hours, Sylgard gels completely, forming a tough, flexible, slightly elastic substance that pulls away from the mold cleanly. Its elastic property permits the Sylgard plate to comply with the surface of the wafer being printed, even if the wafer has an irregular surface. Moreover the Sylgard readily accepts wax or wax solvent mixtures, yet permits easy cleaning in wax solvents. Several printing plates composed of this material were used. A master mold and a Sylgard printing plate are shown in Figures 4 and 5, respectively.

3.3 WAX SELECTION

Early in the project, a single wax was chosen as a masking agent. Due to the wide range of chemical etch reagents - both acid and basic - and highly reactive mixtures that the mask would have to withstand, as well as the local heating effect that sometimes accompanies etching, a high-melting micro-crystalline wax was chosen as the first candidate material. The wax chosen was Multiwax 195M, a hard material melting at 90°C and possessing exceptional chemical inertness. The Multiwax, despite its excellent chemical properties, displayed a low viscosity when molten which allowed the printed wax to spread appreciably after being deposited on the wafer surface, thus blurring the printed pattern and obliterating fine details of the image.

The desire for masking agents with higher viscosities and slightly different physical characteristics led to the investigation of two additional material systems: Apeizon W, a black, vacuum distilled asphalt with a working temperature of about 115°C; and glycol phthalate, a clear yellow, glassy substance with a strongly temperature-dependent viscosity change near its melting point of about 110°C.

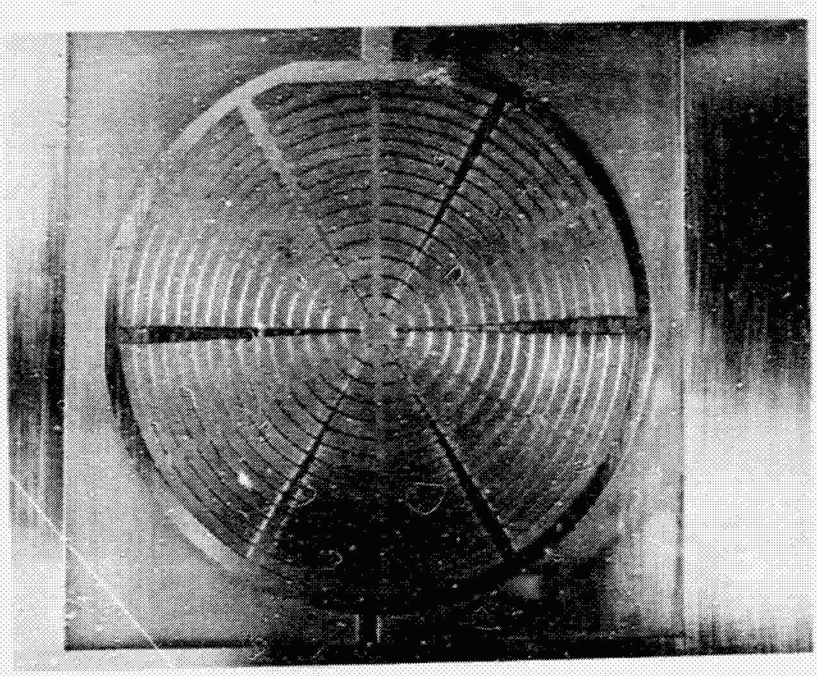


FIGURE 4: STAINLESS STEEL MASTER MOLD (ACTUAL SIZE)

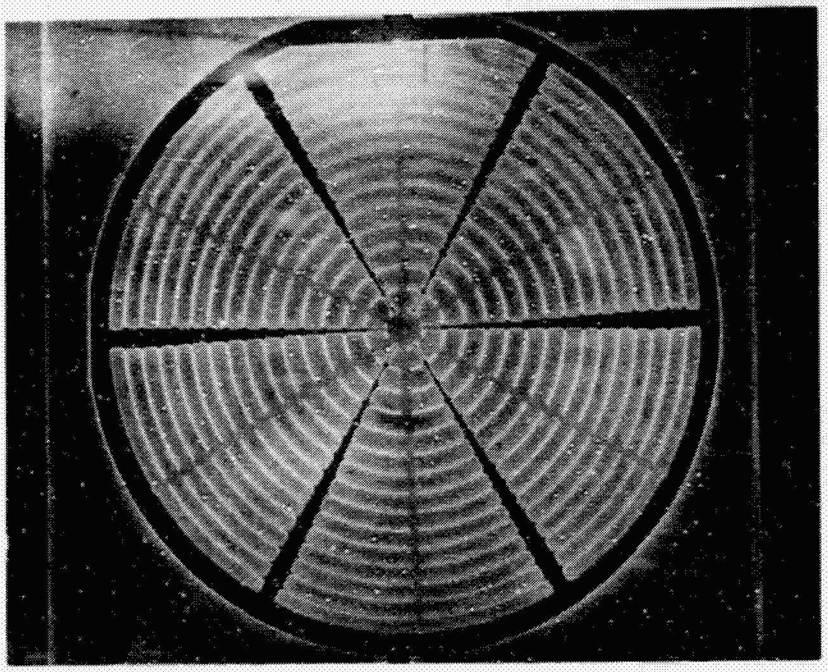


FIGURE 5: SYLGARD PRINTING PLATE (ENLARGED)

Apiezon W was found to be of a high enough viscosity to reduce spreading on polished or etched wafer surfaces and to significantly reduce it on pyramidially textured surfaces. Since Apiezon W is a petroleum-derived substance similar to tar or asphalt, it has good chemical resistance and enough plasticity when cool to allow it to sag into depressions or channels between pyramids on the wafer surface and to stretch to accommodate any wafer expansion or contraction due to temperature changes during processing. In this property also lies the disadvantage of being soft enough to be susceptible to scratching, resulting in possible localized removal of the protective wax layer.

Glycol phthalate, the third wax considered for use, is a hard, clear, glassy substance. It combines hardness (to resist scratches) with a profound viscosity change occurring above its melting point. The viscosity of this substance can thus be altered by control of temperature. The viscosity range available is sufficient to prevent wax spread on any surface. As a disadvantage, the chemical resistance of this wax is only fair.

Table I contains the results of a one hour exposure test of the waxes to concentrated processing reagents at room temperature. While this test imposed conditions more severe than those anticipated in actual processing, it did identify the reagents that were likely to cause erosion of the wax surface, perhaps causing failure of the mask layer. As can be seen, glycol phthalate, despite its hard surface, appears to be damaged by more reagents than the other two waxes, and is generally affected by reagents to a greater extent than the other waxes. This material is best used where physical hardness is important and where acid content of the etching solution is low or strongly buffered, as in texture etching. As anticipated, Multiwax 195M is the least affected wax, with Apiezon giving similar results; these two waxes were chosen for use in the remainder of the project.

TABLE 1: MASKING WAX CHEMICAL RESISTANCE

ETCH REAGENT	WEIGHT LOSS OF WAX SAMPLE IN PERCENT AFTER ONE HOUR AT ROOM TEMPERATURE GLYCOL PHTHALATE	MULTIWAX 195M	APIEZON W
Ammonium hydroxide	-0.6 A,B	--	--
Sulfuric Acid, 96%	8 B	1	-0.6 A,E
Nitric acid, 70%	24 C	--	--
Hydrochloric acid, 37%	0.1 A,B	--	--
Hydrofluoric acid, 49%	--	--	--
Acetic acid, 99%	23	--	--
Aqua regia	1	1 D	-0.6 A
Acetone	100 C	--	--
Isopropyl alcohol	--	--	--
Water	--	--	--
Potassium hydroxide, 45%	--	--	--

NOTES:

A Sample absorbed etchant
B Sample surface turned white
C Sample dissolved completely
D Sample yellowed slightly
E Sample surface shrank and softened

Figures below 1% are not listed except where wax appearance changed.

Negative figures indiate that sample weight increased.

Both of the chosen waxes were demonstrated to adhere to surfaces of bare silicon and to silicon dioxide and silicon nitride.

3.4 PRINTING TESTS

Printing trials were performed on a manually operated press in order to allow the flexibility of operation required to study variations in printing technique. In the trials, a wafer was held horizontally on a vacuum chuck with the side to be printed facing up. Two centimeters above the wafer was the Sylgard printing plate, held in a horizontal position, parallel to the wafer surface, with the wax bearing side of the plate facing down. A lever on the printing device was used to drive the printing plate down to contact the wafer, and to elevate the plate once again after contact. The printing plate was waxed by means of a roller dipping into a pool of molten wax. Provisions were made to heat both the printing plate and the wafer to permit the wax to remain fluid during the print sequence.

The print pattern was one which covered the wafer with wax except for a series of fine concentric circles interconnected by six wide radial spokes.

As originally conceived, the application of patterned etch-resistant coatings to wafer surfaces was to be accomplished by using a printing plate of refractory material to transfer pure molten wax to a heated wafer. Further work pointed out the difficulty of performing this process and suggested a more practical method of operation.

The major difficulty with the original process using molten wax was the necessity of maintaining the wax, the printing plate, and the wafer surface (along with the application rollers and auxiliary equipment) at a temperature above the wax melting point. Although the temperatures needed are modest (about 120°C - 140°C), a fairly long time was required to heat a quantity of wax to this temperature and to attain an even temperature within the wax mass. In addition, temperature limiting is necessary to avoid smoking or charring of the wax. Aside from problems of wax stability, the moving parts of the printing mechanism proved to be difficult to maintain at controlled temperature; inadvertant cooling of the wax caused parts of the printing device to freeze together firmly, and the still relatively hot device parts had to be allowed to cool before manual separation of the parts could be performed. Control of wax viscosity by temperature was further limited because the waxes experienced temperature variations during the printing process, making close viscosity control at the moment of printing very difficult.

An alternate to molten wax is the use of wax-solvent solutions at room temperature. The use of such solutions eliminates the necessity of maintaining an entire printing press at elevated temperature. Additional advantages include the greater control of wax viscosity (by chemical formulation) and greater freedom of selection of printing plate material.

Tests were again conducted, this time using a solution of Apiezon W in methylene chloride, and a suspension of Multiwax 195M in methylene chloride. Use of a suspension in the case of Multiwax was necessary because Multiwax has few true solvents, and those that do dissolve this material either dissolve too little to permit the application of an etch resistant coating (as is the case with mineral spirits), or are not volatile enough to permit the solution to dry to a hard wax coating (as with mineral oil). The printing device was operated at room temperature, the wax was applied to the plate by roller (and printed on wafers as previously described), and the printed wafers were either allowed to air dry or were subjected to a 50°C bake cycle for 30 minutes before acid etching.

3.5 IMAGE FIDELITY

The major problem associated with the process was that of attaining consistent reproduction of the wax mask image. Although the use of special test pl-'es confirmed the ability of the process to produce 2 - 5 mil lines, these results were produced too rarely to warrant claims of a perfected process. Specifically, problems resulted from spreading of wax from its point of application, failure to make firm contact between the printing plate and moderately warped wafers, and the rare but nevertheless frustrating occurances of wafers sticking to the printing plate by means of a "suction-cup" effect. Also, use of the Multiwax suspension was discontinued, since it was noted that the discreet particles of wax in the suspension produced a grainier image than the Apiezon W solutions.

In an effort to improve image fidelity, a cylindrical printing plate configuration was tested. In this variation of the technique, a printing plate was fashioned in the shape of a cylinder which was waxed and then rolled over the wafer surface. Since such an arrangement requires the plate to contact the wafer along a line, rather than over an area, plate-to-wafer contact was improved and suction-cup effects were eliminated. Spreading of wax still resulted.

An analysis of the spreading problem has led to the conclusion that it may be inherent in the process applied to hard substrates. A wax coating, applied to the printing plate in a thickness adequate to ensure transfer of a protective amount of wax to the silicon wafer surface, was squeezed out from between the plate and wafer when contact was made. The analogous process of letterset printing on paper appears highly dependent on substrate (paper) properties. Paper possesses the attributes of flexibility and absorptivity, neither one of which is displayed by the hard and impenetratable silicon of which solar cells are made. The narrowness of the lines which are etched through the passivating oxide or nitride to accommodate the current-collecting metal pattern is necessary to prevent excessive shading of the cell. This narrowness, it was found, virtually assures that the wax applied to adjacent areas will be squeezed out into the thin line region, preventing it from etching in the desired way. Figures 6 and 7 depict details of the line pattern. Figure 6 is a view of the pattern on the original photomask showing the desired line dimensions. Figure 7 shows the corresponding pattern when wax is printed on the wafer. Since wax squeezing is not perfectly regular, this effect cannot be compensated for by using wider lines on the plate.

3.6 WAX REMOVAL

In an attempt to reduce or eliminate the use of organic solvents (and the problems associated with their toxicity and disposal) the project originally intended to develop a solventless removal method for cleaning wafers after the chemical etching step. Also intended was the capability of recovering and recycling the wax.

Early experiments showed that up to 70% of masking wax could be removed from wafers by simply immersing them in boiling water, or in water to which a heavy salt, e.g. potassium sulfate, had been added. In this removal method, wax simply melts and floats to the surface of the water where it can be skimmed off. The addition of a heavy salt raises both the boiling point and the density of the water, accelerating the process.

The early experiments were, of necessity, conducted with wafers coated with thick, continuous layers of wax, most of which was easily removed. Later work, using patterned wafers with thinner coatings of wax demonstrated that the relatively small amount of wax on the wafers melted, but clung to the wafer surface.

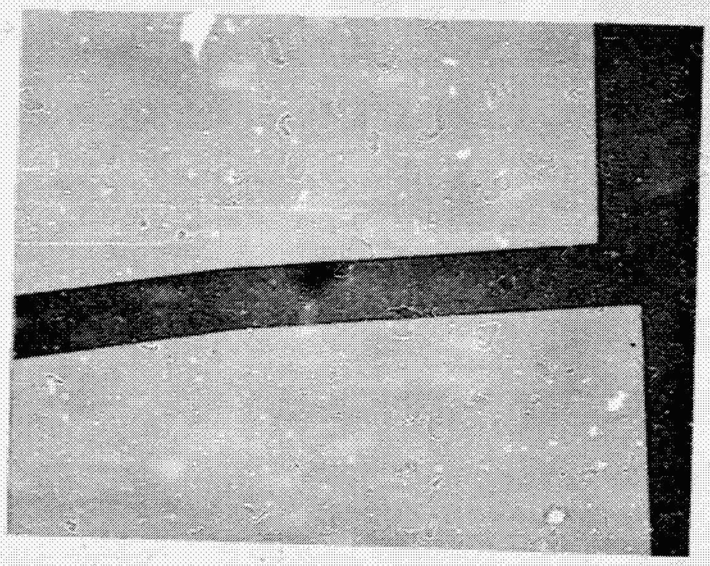


FIGURE 6: DETAIL OF INTENDED PATTERN (LIGHT AREAS ARE TO BE MASKED.) 110X.

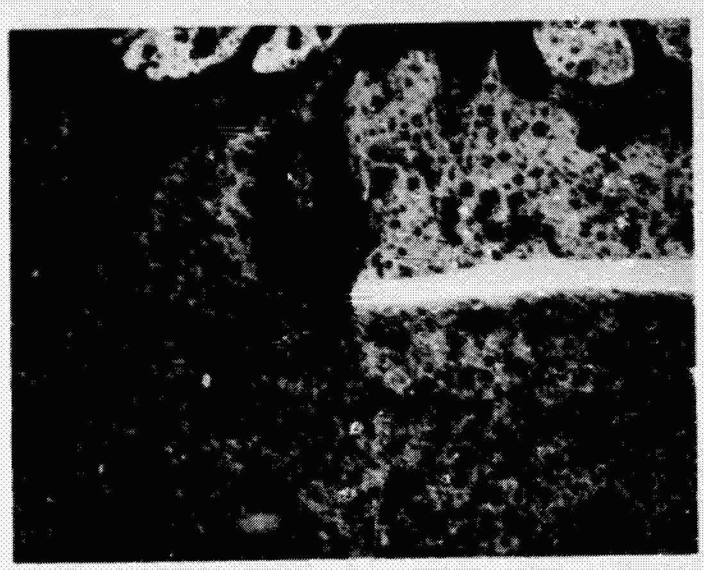


FIGURE 7: DETAIL OF MASKED WAFER SHOWING SPREADING OF WAX. 110X.

Attempts at using blasts of both hot water and steam succeeded in removing more wax, but the removed wax was dispersed as fine droplets in the water and did not separate from the water, so that recovery was impractical. In all cases, enough wax remained on the wafers to make a solvent clean necessary anyway.

The preferred cleaning method was determined to be a solvent clean using perchloroethylene in a degreaser apparatus where the wafers are cleansed by having refluxing perchloroethylene dissolve the wax. Recovery of the wax is still possible with this method by removing perchloroethylene through simple distillation.

4.0 CONCLUSIONS

The conclusions drawn from the results of this project are listed below:

1. Melted wax, e.g. Multiwax 195M, was very resistant to chemical attack by all reagents tested, but required a heated apparatus for deposition; this proved to be too variable for practical use.
2. Wax solutions in the form of Apiezon W or one of its available commercial preparations, could be printed onto silicon substrates, and were found to be useful in masking wafers during wet etching processes.
3. Of the four printing processes investigated (transfer, lithography, intaglio and letterpress), only letterpress demonstrated the possible promise of practical use.
4. The application of patterned wax resist coatings by means of letterpress printing suffers from wax solution squeezing between the plate and the silicon substrate. Cylindrical plates also show this property. Control of this flow will be necessary for practical implementation of the process.
5. No practical alternative to the use of organic solvents for removal of wax was found. Vapor degreasing appears to be the most effective removal process.
6. Recovery of used wax from the residue of a degreaser type cleaning apparatus is possible by standard distillation methods.

5.0 RECOMMENDATIONS

With the effort devoted to this program, a viable alternative to silk screening of appropriate wax formulations was not forthcoming. Additional effort might solve the remaining problems. However, with the advent of an entirely new technology for patterning dielectric layers on silicon, it is questionable whether further effort on wax masking would be desirable.

Demonstrations of fine-line plasma etching of dielectric layers through a reuseable metal mask have already been made. The full development of this process will undoubtedly result in more economic wafer processing, less consumed material, and less disposed waste, than is possible with any wax process that exists today.

6.0 NEW TECHNOLOGY

This project had as its goal the application of existing processes to the manufacture of solar cells. As such, no new technology is reported.